US-PAT-NO: **5453185**
DOCUMENT-IDENTIFIER: US 5453185 A

TITLE: Column with macroporous polymer media

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US-CL-CURRENT: 210/198.2; 210/502.1 ; 210/635 ; 210/656 ; 264/41 ; 264/53
; 502/439

APPL-NO: 08/ 224765

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PARENT-CASE:

CROSS REFERENCE TO RELATED APPLICATION This is a divisional of application Ser.
No. 07/965,181 filed on Oct. 23, 1992 now U.S. Pat. No. 5,334,310, which, in
turn, is a continuation-in-part of U.S. Ser. No. 07/779,911, filed Oct. 21,
1991, now abandoned.

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US PATENT NO. - PN:

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Detailed Description Text - DETX:

After polymerization is complete, the solid macroporous polymer plug is washed
to remove any porogenic solvent and with a suitable solvent to dissolve any
soluble polymer present. Suitable washing solvents include methanol, ethanol,
benzene, toluene, acetone, **tetrahydrofuran,** and dioxan. This washing process
may be done in stages; for example, by washing with a

solvent, then with water
and then a solvent again, or by continuous washing with a solvent. The washing
step is preferably carried out by pumping the solvent through the tube filled
with the macroporous polymer.

Detailed Description Text - DETX:

Still another alternative relating to the washing step involves removing the
polymerized plug from the tube, washing it with a suitable solvent, and then
returning it to the tube. The returned polymer may be left as is or it may be
swelled to occupy more space in the tube by washing the polymer with a swelling
solvent such as **tetrahydrofuran,** 1,4-dioxan, toluene, or halogenated
hydrocarbons.

Detailed Description Text - DETX:

One end of a stainless steel tube (4.6 mm inner diameter, 50 mm length)
provided on both ends with fittings for attachment to a liquid chromatograph
was closed with a steel nut stopper, the tube was purged with nitrogen and its
other end was closed with a silicon rubber septum. A polymerization mixture
was prepared by mixing 4.8 g glycidyl methacrylate, 3.2 g ethylene
dimethacrylate, 10.8 g cyclohexanol, 1.2 g dodecanol, and 0.08 g
azobisisobutyronitrile. The mixture was bubbled with nitrogen for 20 minutes
to remove any oxygen present. 0.1 ml of the mixture was injected through the
septum into the tube, and polymerization was started by heating in an
thermostated oil bath at 70.degree. C. After 7 hours the tube was removed from
the bath, cooled freely to room temperature, and the septum was removed. At
this point the column contained a solid macroporous polymer

plug 5 mm in
length. After washing the polymer with methanol, both ends of the tube were
closed with chromatographic nuts and the column attached to a HPLC
chromatograph. First, methanol was pumped through the column at different flow
rates. The back pressure was 0.4 MPa at a flow rate 0.5 ml/min, 0.8 MPa at 1
ml/min and 1.6 MPa at 2 ml/min. The solvent was then changed to
tetrahydrofuran and the back pressure was 0.2 MPa at 0.2 ml/min and 19 MPa at 2
ml/min. The dependence of back pressure on flow rate was found to be linear.

Detailed Description Text - DETX:

Into the same tube as used in Example I was injected 0.3 ml of a stock mixture
consisting of 3 ml styrene, 1.2 ml divinylbenzene (85% of DVB), 6 ml
benzylalcohol, and 0.05 g benzoylperoxide. After degassing, the tube was
closed and polymerization proceeded at 70.degree. C. for 24 hours. Using a
steel plunger an 18 mm long porous polymer plug was pushed out of the tube and
washed for 4 days in several portions of methanol. After the washing the plug
still fit in the original tube. When the solvent was changed to
tetrahydrofuran, it swelled the plug. After about 20 minutes, the diameter of
the plug exceeded the diameter of the tube. In order to shrink the plug to its
original size, methanol was used for subsequent exchange of the solvent. The
plug was then returned to the column and methanol was again exchanged back to
tetrahydrofuran. The swelling in the column was monitored as a change of back
pressure which increased from 0.3 MPa at the beginning when the rest of the
tube was still filled with methanol to more than 10 MPa after exchanging some
of the solvent. At that point the measurement was

terminated to prevent any
possible damage of the equipment.

Detailed Description Text - DETX:

The procedure of Example 1 is repeated except that the polymerization mixture
is replaced by the formulation of Dutch patent application No. 6,803,739,
example II, the tube is of flexible
polytetrafluoroethylene, and the
polymerization temperature is 60.degree. C. When the resulting tube and with
its polymer plug is connected to a conventional
chromatograph, essentially no
flow of **tetrahydrofuran** through the plug is observed at pressures as high as 40
MPa (6,000 psi). When the plug is evaluated for pore size distribution as in
Example 6 by cutting the tube apart to remove the plug, no pores larger than
about 200 nm are found.